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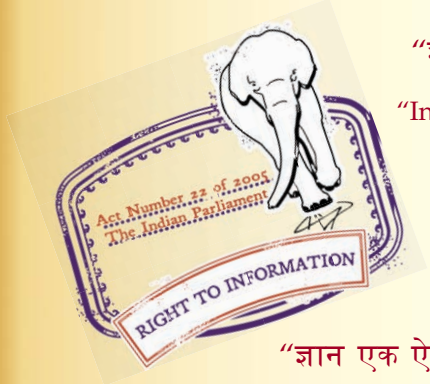
IS 11962 (1987): Method for determination of ash in rennet caseins and caseinates (refernce method) [FAD 19: Dairy Products and Equipment]

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"Knowledge is such a treasure which cannot be stolen"

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**IS 11962 : 2012**
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*भारतीय मानक*

# रैनेट केसीन एवं केसीनेटस — राख निर्धारण (संदर्भ पद्धति)

*( पहला पुनरीक्षण )*

*Indian Standard*

# RENNET CASEINS AND CASEINATES — DETERMINATION OF ASH (REFERENCE METHOD)

*( First Revision )*

ICS 67.100.10

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**BUREAU OF INDIAN STANDARDS**
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NEW DELHI 110002

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Dairy Products and Equipment Sectional Committee, FAD 19

NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical with ISO 5545 : 2008 'Rennet caseins and caseinates — Determination of ash (Reference method)' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dairy Products and Equipment Sectional Committee and approval of the Food and Agriculture Division Council.

This standard was first published in 1987 under dual numbering system which was identical with ISO 5545 : 1978. This first revision has been brought about to align it with the latest version of ISO 5545 : 2008 which has been technically revised.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

| *International Standard* | *Corresponding Indian Standard* | *Degree of Equivalence* |
|---|---|---|
| ISO 3310-1 Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth | IS 460 (Part 1) : 1985 Specification for test sieves: Part 1 Wire cloth test sieves | Technically Equivalent |
| ISO 5550 : 2006 Caseins and caseinates — Determination of moisture content (Reference method) | IS 11920 : 2012 Caseins and caseinates — Determination of moisture content (Reference method) (*first revision*) | Identical |

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard*

# RENNET CASEINS AND CASEINATES — DETERMINATION OF ASH (REFERENCE METHOD)

*( First Revision )*

## 1 Scope

This International Standard specifies a reference method for the determination of the ash of caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate.

NOTE For the determination of ash ("fixed ash") of acid caseins, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type, see ISO 5544 | IDF 89 [2].

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 5550 | IDF 78, *Caseins and caseinates — Determination of moisture content (Reference method)*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

**3.1**
**ash of rennet caseins or of caseinates**
substances determined by the procedure described in this International Standard

NOTE Ash of rennet caseins or of caseinates is conventionally expressed as a percentage by mass, numerically equivalent to grams per 100 g of product.

## 4 Principle

A test portion is incinerated at (825 ± 25) °C. The residue is then weighed.

## 5 Apparatus

Usual laboratory apparatus, and in particular the following.

**5.1** **Analytical balance**, capable of weighing to the nearest 0,000 1 g.

**5.2** **Silica** or **platinum dishes**, of diameter about 70 mm and of depth 25 mm to 50 mm.

**5.3 Electrical furnace**, with air circulation, capable of being maintained at (825 ± 25) °C.

**5.4 Desiccator**, containing an effective desiccant.

**5.5 Grinding device**, for grinding the laboratory sample, if necessary (see 7.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

**5.6 Test sieve**, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310-1.

## 6 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50 [1].

## 7 Procedure

### 7.1 Preparation of the test sample

**7.1.1** Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an airtight container of sufficient capacity to allow this operation to be carried out).

**7.1.2** Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (5.6).

**7.1.3** If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 7.1.1.

**7.1.4** Otherwise, grind the 50 g portion, using the grinding device (5.5), until it passes the sieve. Immediately transfer all of the sieved sample to an airtight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

**7.1.5** After the test sample has been prepared, the determination (7.4) should proceed as soon as possible.

Clean the device after grinding each sample.

### 7.2 Preparation of the dish

Heat the dish (5.2) in the electrical furnace (5.3), maintained at 825 °C, for 30 min. Allow the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1 mg.

### 7.3 Test portion

Weigh, to the nearest 0,1 mg, directly in or by difference approximately 3,000 0 g of the test sample (7.1) into the prepared dishes.

### 7.4 Determination

Heat the dish containing the test portion on a low flame until the test portion is completely charred, taking care that it does not burst into flame.

Transfer the dish to the electrical furnace (5.3), maintained at (825 ± 25) °C, and heat for at least 1 h until all carbon has disappeared from the dish. Allow the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1 mg.

Repeat the operations of heating in the electrical furnace (5.3), cooling and weighing, until the mass remains constant to within 1 mg or begins to increase. Record the minimum mass.

# 8 Expression of results

## 8.1 Calculation

**8.1.1** The ash of the sample, $w_a$, as a percentage by mass, is given by Equation (1):

$$w_a = \frac{m_1 - m_2}{m_0} \times 100 \quad (1)$$

where

$m_0$ is the mass, in grams, of the test portion (7.3);

$m_1$ is the mass, in grams, of dish and residue (7.4);

$m_2$ is the mass, in grams, of the prepared dish (7.2).

Calculate the ash to the nearest 0,01 % by mass and report the final result to the nearest 0,1 % by mass.

**8.1.2** To calculate the ash of the sample on the dry basis, as a percentage by mass, multiply the result obtained from Equation (1) by Factor (2):

$$\frac{100}{100 - w_w} \quad (2)$$

where $w_w$ is the water content, as a percentage by mass, of the sample determined according to ISO 5550 | IDF 78.

## 8.2 Precision

### 8.2.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time will in not more than 5 % of cases be greater than 0,15 g of ash per 100 g of product (numerically equivalent to 0,15 % by mass).

### 8.2.2 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,25 g of ash per 100 g of product (numerically equivalent to 0,25 % by mass).

## 9 Test report

The test report shall specify:

a) all the information required for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, together with a reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s);

e) the test result(s) obtained, and if the repeatability has been checked, the final quoted results obtained.

# Bibliography

[1] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*

[2] ISO 5544 | IDF 89, *Caseins — Determination of "fixed ash" (Reference method)*

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This Indian Standard has been developed from Doc No.: FAD 19 (1962).

### Amendments Issued Since Publication

| Amendment No. | Date of Issue | Text Affected |
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